

TUBE WITH HEAD MADE OF MULTILAYER MATERIALS  
AND MANUFACTURING PROCESS

## Field of the invention

The invention relates to the domain of plastic tubes, and particularly tubes with barrier properties, typically for toothpaste tubes.

5 State of the art

European patent EP-B-0524 897 submitted in the name of the applicant discloses a process for the manufacture of tubes in which firstly, particularly in order to improve the barrier effect of tube heads, a separate single part or insert made of a 5-layer multilayer material PE/adh/EVOH/adh/PE is prepared by thermoforming, where "PE" denotes a polyethylene layer, "EVOH" denotes a vinyl ethylene alcohol copolymer layer with a barrier effect, "Adh" denotes an adhesive layer to assure cohesion between the PE and EVOH layers,

A tube skirt and the said insert are then placed on a punch of an insert molding tool, the two being manufactured separately, and the PE head is then insert molded to obtain a tube as shown in figure 1.

Documents AU-0545 604 and EP-A-0130 239 describe other processes for manufacturing tubes including the use of inserts, typically polybutyleneterephthalate (PBT) inserts made separately by molding.

25 Finally, document BE-A-666 719 describes a tube in which the head comprises an insert in the form of a truncated cone made of a folded thin sheet of aluminum, as barrier material.

Statement of the problems

Problems can arise with known tubes according to standard practice, as a result of using inserts made separately:

5       - firstly, separate manufacturing of an insert, particularly in the case of an insert with five layers as described in document EP EP-B-0524 897, is an additional operation, for which the manufacturing cost is additional to the cost of an identical tube without  
10       an insert, plus the assembly cost of the insert itself and the increased complexity of assembly machines,

15       - secondly, the inserts form barrier materials and therefore with a different nature from the PE. Since they are made separately, they add a non-negligible percentage to the weight of the tube, and insert materials (particularly PBT) are immiscible with PE which is the typical material used in tubes, introducing a problem with recycling the materials used in the tubes,

20       - finally, the inserts often create a barrier problem, either because the intrinsic barrier properties of the barrier material are inadequate, or because there is an excessive annular band at the connection zone between the skirt and the head.

25       The heads and tubes according to the invention solve all these problems simultaneously.

Description of the invention

30       According to a first purpose of the invention, the plastic tube head, designed to be assembled with a skirt to form a tube, comprising a typically threaded

orifice and a shoulder comprising an annular connection part to the said skirt, is characterized in that:

5 a) the said head is formed by co-injection and comprises a multilayer material containing an internal layer, an external layer in structure material, typically a polyolefine, and at least one inner layer made of a thermoplastic barrier material,

10 b) the said inner layer is encased by the said internal and external layers, including at the ends of the said head where the said internal and external layers are joined together in a polyolefine layer, the distances "e" and "e'" between each of the ends of the said inner layer and the corresponding end of the said head being between 0.02 mm and 5 mm and preferably less  
15 than 3 mm, such that the said inner layer made of a barrier material extends over the greatest possible height, while its ends remain encased or encapsulated by the junction of the said internal and external layers.

20 The invention introduces a radical change in the design of the said head, firstly in the sense that the said internal layer is completely immersed and encased by a so-called structure material A, typically a polyolefine material - but it may also be another  
25 extrudable material such as a PET or a PA - without discontinuity or break in the material flow as is the case with the use of an insert according to the prior art, and secondly in that the said inner layer may be extended as far as required and as close as required to  
30 the ends of the said head, the only limit being that this layer is not in contact with the outside and remains encased by the structure material A.

Since there are no inserts or add-on parts, the tube according to the invention has several important advantages for persons skilled in the art, concerning the cost, the compatibility of the head and skirt materials and therefore recycling, since a typical insert is made of PBT, a unique material that does not exist in the skirt, and the fact that the performance of the head can be adapted depending on needs, knowing that not all packaged products have the same requirements concerning impermeability of the head, and therefore that the nature of the barrier layer and its thickness can be varied to obtain tubes adapted to needs at minimum cost.

Said barrier layer may be formed from one or more layers which overlap at least in part.

#### Description of the figures

Figures 1a and 1b are axial sectional views of a tube head 2 according to the state of the art provided with an insert 4. This head has an orifice 20 in which a thread 201 is formed, and a connection shoulder 21 to a skirt 3.

Figure 1a shows a view of a head 2 with its injection gate 26 at the exit from the insert molding station where the head 2 is molded on the skirt 3.

Figure 1b corresponds to the head of the tube 2 in figure 1a after the injection gate 26 has been eliminated and after the orifice 200 has been formed.

Figure 1b shows an enlarged view of the section through the skirt 3 that typically comprises a layer made of a barrier material B coated with an external

layer 31 and an internal layer 32 in structure material A.

Figures 2a and 2b correspond to figures 1a and 1b and to the invention. The material forming the head 2 comprises an inner layer made of barrier material B 25 coated on the outside with an outer layer 23 and on the inside with an internal layer 24 in structure material A.

Figure 2b shows distances denoted "e" and "e'" between the ends of the said internal layer 24 and the ends of the head.

Figure 3 shows a vertical sectional view of a co-injection nozzle 5 with a vertical axis of symmetry 10 having a central duct 50, an outer ring duct 52 for injection of material A, a median ring duct 51 for injection of barrier material B and a common exit opening 53.

Figure 4 contains a vertical section showing a diagrammatic view of an individual co-injection device. This device comprises a nozzle 5 supplied firstly by material A (typically PE) via an injection device for material A comprising upstream a supply channel 633 fitted with a valve or closing slide 635, a proportioning piston 632 supplied with material A from an extruder 63 and fitted with a non-return valve 631, and secondly with barrier material B by a barrier material B distributor comprising a supply channel 643 fitted with a valve or closing slide 644 and, upstream, an extruder 64 of barrier material B.

The nozzle 5 comprises a shutter 65 for its common opening 53 and terminates in an external opening 66 cooperating with the head mold tooling 2 by injection.

The nozzle could comprise axial shutter means (not shown) typically slide means, with which to shutter the exit of material A, that of material B or both materials A and B at the same time.

5       The mold tooling typically comprises a die 61 and a punch 60 on which a tube skirt 3 has been placed, the space between the die and punch forming cavity 67 in which said head 2 is formed by coinjection. The piston 62 applies the punch plus die assembly in contact with  
10   the said external opening 66 at a sufficient pressure to inject the said head 2.

In this figure the distributors of material A and material B for the simultaneous production of several tube heads are not shown.

15       Figures 5 and 6 show another modality of the process and device according to the invention.

Figure 5 is a principle diagram showing in axial section a co-injection device for structure material A (on the left side of the figure) and for material B (on  
20   the right side of the figure). This figure shows the distributors of material A 630 and of material B 640, intended to feed six cavities 67 simultaneously. The supply circuit for material A comprises an injection piston 632 and a non-return valve 631 used to supply  
25   the co-injection head 6 with material A at the required flow rate and pressure.

The device comprises a closing slide 65 with a slide 650 having 4 positions through side movement of the slide 650 which is supplied either with multilayer  
30   material A and B via opening 53 of nozzle 5, or with material A only via the side duct 634 for material A which leads either to cavity 67 or to a drain 68. Slide

650 is shown in cross-section in figures 5 and 5b and in longitudinal section in figure 5a in which the 4 positions are shown:

Position 1: the slide is full, so that none of  
5 inlets 53, 634 and none of the outlets towards the  
cavity 67 or the drain 68 communicate.

Position 2: the arrival of material A, 634, is made to communicate with cavity 67, the arrival of multilayer material A and B via opening 53 being shuttered,

Position 3: the arrival of multiplayer material A and B via opening 53 of nozzle 5 is made to communicate with cavity 67, the arrival of material A, 634, being shuttered.

15            Position 4: the arrival of multiplayer material A  
and B via opening 53 of nozzle 5 is made to communicate  
with the drain 68, a position which is typically used  
not during the production cycle, but in the event of  
stoppage of the said device before re-start up of the  
20    production cycle.

Figure 6 is a perspective view of the distributors of material A, 630, and material B, 640, respectively supplied by the extruders of material A, 63, and of material B, 64, which feed the 6 co-injection heads 6 each comprising a nozzle 5 and a side duct 634 forming the two inlets to the slide valve 65. This figure does not show the 6 injection pistons for material A, 632. This figure shows the arrangement of the slide valves 65 each having two "inlets" or feed points 53 and 634, and two "outlets" 66 towards cavity 67 and drain 68, these "inlets" and "outlets" forming a cross in a plane perpendicular to the axis of the valve 65.

Figure 7 is a diagrammatic overhead view of a tube manufacturing unit with six injection nozzles 5.

Figure 7a diagrammatically shows a sectional view of a skirt 3, and figure 7b shows a co-injection output tube with a head corresponding to figure 2a.

Figure 8 illustrates the execution of an injection cycle with a total duration of  $T_0 + T'$ , injection of structure material A (typically a polyolefine of PE type) through valve 635 varying from time  $T=0$  to  $T=T_0$ , whereas the injection of barrier materials B through the valve or slide 645 starts at time  $T=t$  and terminates at time  $T=T_0 - t'$ , the duration  $T'$  being a stabilization time.

The axis denoted "P650" shows the position of slide 650 when the device used comprises this type of 4-position slide, designated 1 to 4, as shown in figures 5a and 5b.

#### Detailed description of the invention

The said internal layer 24 and external layer 23 in head 2 of tube (1 according to the invention may be made of the same polyolefine, preferably chosen to be a polyethylene or PE, and the said barrier material may be chosen from known extrudable barrier materials and is preferably chosen to be a polyvinyl alcohol or EVOH, but as already indicated the invention is not limited to specific materials, other than that these materials must be extrudable or co-extrudable.

Moreover, it is also possible according to the invention to have as structure material a material A for the external layer 23 and a material A' different to the previous one for the internal layer 24.



Preferably, the said multilayer material has a 3-layer "A/B/A" structure, where A and B respectively denote a structure material layer, typically a polyolefine and preferably PE - that is the internal layer 24 and external layer 23, and a barrier material layer - that is the inner layer (25) typically made of EVOH, the layer of barrier material B having an average thickness of between 0.02 and 0.5 mm, and preferably an average thickness of 0.04 and 0.2 mm, for a total thickness of the said 3-layer structure typically equal to 1.2 mm.

It was observed that it is not necessary, according to the invention, to insert an adhesive layer between the structure material layer A and the barrier material B, even though materials A and B may not adhere to one another, as is the case with PE and EVOH.

Also, layer B may optionally be replaced by several B layers of narrower thickness, which overlap in full or in part, possibly with layers A inserted in between.

As already described, the thickness of the barrier material layer may be varied as a function of the required performances, considering the nature of the packaged product.

Another purpose of the invention consists of the tubes 1 that comprise a head 2 according to the invention.

Preferably, the assembly of the said skirt and the said head is formed by co-injection of the said head on the said previously formed skirt.

Although the heads can be made separately and then assembled with the skirts, it is advantageous to

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proceed as shown in figure 4, in other words forming the head by injection on a skirt, combining the formation of the head and the head and skirt assembly in a single step, the skirt possibly being manufactured  
5 apart.

Another purpose of the invention consists of a process for manufacturing a multilayer head according to the invention.

In this process:

10 a) into a cavity 67, formed by the cooperation of a die 61 and a punch 60 and using a co-injection head 6 supplied with structure material A and barrier material B, a flow of structure material A is co-injected for a time  $T = T_0$  with, from a time  $T = t$  until a time  $T =$   
15  $T_0 - t'$ , a flow of barrier material corresponding to said inner layer 25, times  $t$  and  $t'$  being chosen as short as possible such that, after the injection gate 26 has been eliminated, the ends 250, 251 of the said inner barrier material layer 25 of the said head 2 are  
20 entirely encapsulated by the said structure material A of the said internal and external layers 24, 23, the said ends 250, 251 being separated from the external medium by a thickness of at least 20  $\mu\text{m}$  of the said structure material A,

25 b) injection of the said structure material A is continued for an additional time  $T'$  equal to at least  $T_0$ , in order to stabilize the quantity of injected structure material.

Typically:

- $T_0$  can vary from 0.1 s to 1 s
- 30 -  $T_0 + T'$  can vary from 1 s to 3 s
- $t$  can range from 0.01  $T_0$  to 0.5  $T_0$
- $t'$  can range from 0.01  $T_0$  to 0.65  $T_0$

This process will be better understood with reference to figures 3, 4, 5, 5a, 5b and 8.

It can be clearly seen in figure 8 that the injection of the barrier material B begins after the injection of structure material A and terminates before the injection of structure material A.

Thus, it is clear that with all means defined by the invention, it is possible during a full head production cycle T, to regulate the introduction of each injected material and thus to control both distances "e" and "e'".

Furthermore, these means can also modify the thickness of the barrier material layer B at will, by varying the relative flows of the barrier material B and structure material A in the co-injection nozzle 5.

A further purpose of the invention is a process for manufacturing tubes having a head 2 according to the invention.

According to a first embodiment, a head according to the invention can be assembled to the skirt by any known means, typically by any type of weld. But preferably, said head and said skirt are assembled by co-injecting the said head on the said skirt as shown in figure 4.

A manufacturing unit using the process according to the invention is shown diagrammatically in figure 7. In this industrial process a turntable or carousel 76 with a vertical axis of rotation 77 divided into p sectors 71, 72, 73, 74 where p is typically equal to 8 and is indexed in rotation with an angular pitch equal to  $360^\circ/p$ , brings each sector successively in front of at least three fixed stations staggered at an angle

relative to said axis of rotation, that is a first skirt loading station 71 on the said sector of the turntable, then a second station 72 for co-injection and insert molding of the said heads on the said skirts, and a third section at which the tubes 74 are unloaded from the said turntable, the residence time of a sector facing each of the fixed stations being equal to the sum  $T_o+T'$ , preferably varying from 1 second to 3 seconds, and the time interval between two fixed stations being determined particularly by the angular offset between these two fixed stations.

Figure 7 shows the case in which, with  $p$  equal to 8, the angular offset between the co-injection station 72 and the unloading station 74 is equal to  $\alpha$ , typically equal to  $180^\circ$ , such that the tube cooling time between the co-injection station and the unloading station is approximately equal to  $(T_o+T') \cdot (p/360^\circ) \cdot \alpha$ .

#### Example embodiment

All figures (except figures 1a and 1b) are related to the invention and illustrate the invention.

The tube heads obtained according to the examples are shown in figures 2a and 2b.

The dimensions of these heads are standard dimensions of toothpaste tube heads, the height of the head being 20 mm and the tube diameter being 35 mm.

The materials used are PE for the structure material, and EVOH for the barrier material.

The thickness of the multilayer material at the shoulder 21 is typically 1.2 mm, and the thicknesses of the PE layers 23, 24 and the barrier material layer 25 are 1 mm for the outer layer of PE 23, 0.05 mm for the

inner layer of EVOH 25 and 0.15 mm for the internal PE layer 24.

The distances "e" and "e'" obtained for the heads are equal to 2 mm.

5        These heads are made using co-injection nozzles 5 as shown on figure 5.

      This type of nozzle 5 is supplied with PE via duct 633 and with EVOH via duct 634, and comprises means for regulating and opening/closing said ducts.  
10       Since the quantity of PE is much greater than the quantity of EVOH, each nozzle 5 comprises a proportioning piston 633 that injects a precise quantity of PE into the said cavity - a non-return valve 631 preventing any backflow of PE. This  
15       proportioning piston 633 is filled during idle time while the tube is being changed.

      Nozzle 5 has an orifice 53 which is automatically closed by a non-return valve 65 when the cavity 67, formed by cooperation of punch 60 with die 61, is not -  
20       by means of piston 62 - applied against the external orifice 66 for the purpose of co-injecting PE and EVOH.

      According to one variant of the invention shown in figures 5 to 6, nozzle 5 is only used for co-injecting materials A and B, the injection of material A alone  
25       being made via a specific duct 634 which does not cross through the nozzle 5, the changeover from one injection type to another being made by means of a slide valve 65 with a slide 650 which moves sideways and has 4 positions as explained in relation to figures 5, 5a, 5b  
30       and 6.

      The sequence of a production cycle for a head 2 is illustrated in figure 8.

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The duration of a cycle  $T_0+T'$  varies from 1 s to 3 s, and is typically close to 2 s.

In order to guarantee good productivity, the co-injection station on the production line used to implement the process according to the invention comprises six nozzles 5 in parallel, supplied with material A via ducts or supply channels 633, and with material B via channels 643. The co-injection head 6 in figure 6, in addition to direct supply from nozzle 5 of material A via channel 633, also comprises a supply of material A via side channels 643.

In this latter case, in which the co-injection device comprises a parallel supply of material, and in which a slide valve 65 is used with a 4-position slide 650, the line "P650" in figure 8 shows the operation during two production cycles, each one lasting a time  $T_0+T'$ .

Time  $T_0$  comprises the cycle change, the slide being in position "1", in which there is no flow of material, this cycle change corresponding to the replacement of the full cavities by empty cavities, and is conducted during a time interval that is typically less than 0.1  $T_0$ .

The industrial production line is shown diagrammatically by the overhead view in figure 7. It comprises a carousel 76 or turntable rotating around its vertical axis 77 divided into 8 sectors that pass in sequence in front of a first station 71 supplying skirts 3, then in front of a second co-injection station 72 offset at an angle of  $90^\circ$  ( $360^\circ \cdot 2/8$ ) from the first, then in front of a third unloading station 74 at an angle of  $270^\circ$  from the first station, such

that the tube head cooling time on the carrousel is equal to approximately  $4 \cdot (T+T')$ .

The productivity of this line is about 10 000 tubes per hour, substantially equal to  $6 \times 3600 / (T_0 + T')$ ,  
5 where  $T_0 + T'$  is close to 2 s.

#### Advantages of the invention

The invention provides an advantageous alternative to the state of the art. As already mentioned, the  
10 invention is a means of avoiding the use of inserts, which are made separately from a special material and which act as a barrier material.

Therefore, the invention can solve all problems related to the presence of inserts, whether in respect  
15 of the compatibility of materials and recycling of tubes, the extension of the insert and its barrier effect over the entire height of the head, the adaptation of the barrier in relation to the content and conditions of use, or finally in respect of the  
20 cost of the tubes.

The invention is a means of obtaining EVOH layers as thin as possible and as desired in relation to required barrier impermeability, whereas an insert  
needs to have minimum stiffness and therefore a minimum  
25 thickness owing to handling requirements and since it must be manufactured separately.

Therefore, the invention provides a very general means to enable a tube manufacturer to meet most requirements, particularly regulatory and legislative  
30 requirements, related to tube materials and their recycling.

## LIST OF REFERENCES

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